MEMORANDUM

SUBJECT: Glyphosate Trimesium in or on Corn, Soybeans, Citrus Fruit, Stone Fruit and the Nut Crop Group (except Almonds). (MRID#s: 432736-04 and -05. Barcode D216509. Chemical No 128501. CBTS# 15715.)

FROM: Douglas Swineford, Chemist
Analytical Chemistry Section

THRU: Harvey K. Hundley, Head
Analytical Chemistry Section

THRU: Donald A. Marlow, Chief
Analytical Chemistry Branch
Biological and Economic Analysis Division (7503W)

TO: F. D. Griffith, Acting Section Head
Tolerance Petition Section III
Chemistry Branch I, Tolerance Support
Health Effects Division (7509C)

Introduction

The Analytical Chemistry Laboratory (ACL) validated the method: "Determination of Residues of the Trimethylsulfonium Cation in Agricultural Crops by Gas Chromatography" (Study Number GLYP-93-AM-04/Document Number RR93-105B). The matrices validated were pecans, corn grain, corn fodder, soybean seed and oranges.

Method Summary

The samples were extracted with an aqueous phosphate buffer and filtered. The extract is treated with basic anion-exchange resin to neutralize acids followed by phenylisocyanate and barium hydroxide to remove amino acids. The samples were cleaned up with cation-exchange resin and dealkylated prior to quantitation by gas chromatography (GC) using a flame photometric detector (FPD).
Comments

1. Two experienced chemists can process a set of six samples in 12 hours, excluding the optional thermal cleanup (sec. 3.5) that may be necessary for some matrices. GC analysis time is approximately 5 minutes per injection.

2. The limit of detection from visual inspection of the chromatograms is estimated to be 0.01 ppm for the matrices tested.

3. No special safety hazards were noticed. Normal laboratory safety procedures were followed.

4. Standards were obtained from the registrant (Zeneca Ag Products, Richmond, CA 94840-0023). The EPA standards repository in RTP, North Carolina indicated they have a small amount of the standard available.

5. ACL has received various analytical methods this year for the validation of the trimethylsulfonylum cation from Zeneca Ag Products for multiple raw agricultural commodities. Has the registrant considered the feasibility of combining the various approaches into a single method?

6. If the above comments are taken into consideration and incorporated with additional comments noted in the laboratory pre-review, the method would meet 40 CFR 158 and EPA's requirements as published in the Pesticide Assessment Guidelines, Subdivision "O" for Residue Chemistry, Part 171-4(b) as an enforcement method.

7. Recovery data, example chromatograms, pre-review checklist and pre-review comments are attached.
# Validation Report for Trimethylsulfonium Cation (TMS)

<table>
<thead>
<tr>
<th>Commodity</th>
<th>Chemical Added</th>
<th>PPM Added</th>
<th>PPM Found</th>
<th>% Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Corn Grain</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
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<td>0</td>
<td>N.D.*</td>
<td></td>
<td>---</td>
</tr>
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<td>N.D.*</td>
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</tr>
<tr>
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<td>0.0463</td>
<td>92.6</td>
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* N.D. = < 0.01 ppm

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<th>Chemical Added</th>
<th>PPM Added</th>
<th>PPM Found</th>
<th>% Recovery</th>
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<tr>
<td><strong>Corn Fodder</strong></td>
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N.D.* = < 0.01 ppm
### VALIDATION REPORT TRIMETHYLSULFONIUM CATION (TMS) (continued)

<table>
<thead>
<tr>
<th>Commodity</th>
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<th>PPM Added</th>
<th>PPM Found</th>
<th>%Recovery</th>
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<td>N.D.*</td>
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* N.D. = < 0.01 ppm

<table>
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<th>Commodity</th>
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<th>PPM Added</th>
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<td>N.D.*</td>
<td>---</td>
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* N.D. = < 0.01 ppm
<table>
<thead>
<tr>
<th>Commodity</th>
<th>Chemical Added</th>
<th>PPM Added</th>
<th>PPM Found</th>
<th>% Recovery</th>
</tr>
</thead>
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<tr>
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<td>N.D.*</td>
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<tr>
<td></td>
<td>Control</td>
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<td>N.D.*</td>
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</tr>
<tr>
<td></td>
<td>TMS</td>
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<td>TMS</td>
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<td>0.1026</td>
<td>103</td>
</tr>
</tbody>
</table>

* N.D. = < 0.01 ppm
Modifications to method:

None.

Special precautions to be taken:

Handle all chemicals in a safe manner.

Source of analytical standard:

Zeneca Ag Products, Richmond, CA 94804-0023.

If derivatized standard, give source:

N/A

Instrument for quantitation:

GC/FPD

Instrument for confirmation:

N/A

If instrument parameters differ from those given in method, list parameters used:

N/A

Commercial sources of any special chemicals or apparatus:

N/A

Additional comments:

See report.

Chromatograms:

Copies attached.
TMV Pre-Review of Glyphosate - Trimesium

Reviewed By: Dallas Wright, Jr.  

Date: June 15, 1995

Laboratory Assignment Number: B95-34,36,37,38,39

Analyte: Trimethylsulphonium Cation

Commodities: Pecans, Corn grain, Soybean Hay, Soybean Seed, and Oranges

Petitioner: Zeneca Ag Products


Section 3: Analytical Procedure

3.2.2: In the note for this step there are suggestions for filtering the extract; however, there is no filtration step in the extraction for oily crops.

3.3.5: Is it necessary to continue the evacuation of the flask for 1-2 minutes as in step 3.3.4? Should the filter cake be removed before the second filtration?

3.5 Thermal Cleanup: Normally ACL safety regulations do not allow overnight heating or extractions. However, this step should be included in the validation. Is it possible to reduce the time to 8 or 9 hours?

3.6 Dealkylation: 1) Since the toluene is added before the sample is heated, it is possible that the volume will change due to leaking during the heating. Since there is only 0.5 to 1 ml of toluene to start with, a small loss during heating could result in a large percentage change to the final volume used for calculations.

Section 5: Calculations: The instructions are misleading and seem to indicate that it would be appropriate to use the average response factor when using an FPD. The average response factor method for calculating cannot be used when the samples are quantitated on an FPD in sulfur mode, since the response is not linear. The method should clearly state that the analyst must use either a linear regression based on the square root of the response or closely match standard and sample responses.

Section 8.4 Lower Limit of Quantitation: 1) The LOQ for this method is 0.05 ppm for food crops and 0.1 ppm for feed items.
Section 9 Conclusions: The method has not been tested for samples less than the LOQ and greater than 10 times the LOQ. Some of the requested spiking levels are greater than 10 times the LOQ. The following table summarizes the data for each commodity requested, the LOQ, and the requested spiking levels:

<table>
<thead>
<tr>
<th>Commodity</th>
<th>LOQ (PPM)</th>
<th>10x LOQ (PPM)</th>
<th>Data Provided (PPM)</th>
<th>Requested Levels (PPM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pecans</td>
<td>0.05</td>
<td>0.5</td>
<td>0.05; 0.1</td>
<td>0.05; 0.10</td>
</tr>
<tr>
<td>Corn Grain</td>
<td>0.05</td>
<td>0.5</td>
<td>0.05; 0.5</td>
<td>0.05; 0.10</td>
</tr>
<tr>
<td>Corn Fodder</td>
<td>0.1</td>
<td>1.0</td>
<td>0.1; 1.0</td>
<td>0.1; 1.0; 2.0</td>
</tr>
<tr>
<td>Soybean Seed</td>
<td>0.1</td>
<td>1.0</td>
<td>0.05; 0.1</td>
<td>0.05; 0.5; 1.0</td>
</tr>
<tr>
<td>Oranges</td>
<td>0.05</td>
<td>0.5</td>
<td>0.05; 0.09; 0.23</td>
<td>0.05; 0.5; 1.0</td>
</tr>
</tbody>
</table>

Recovery Data: Not all recovery data was within the range of 70-120%. The following list summarizes the data: Pecans: 62-112%; Corn grain: 81-118%; Corn Fodder: 85-115%; Soybean seed: 74-110%; Oranges: 104-125%

ILV Data: The Independent Laboratory Validation was run by Morse Laboratories, Inc. on corn grain (0.05 and 0.5 ppm), forage (0.1, and 1.0 ppm), and fodder (0.1, and 1.0 ppm). Acceptable recoveries were obtained on the first set of samples run.
ANALYTICAL CHEMISTRY BRANCH
SCREEN FOR RESIDUE METHODS FOR TMV

1. LABORATORY ASSIGNMENT NUMBER: B95-34:36-39
2. PP#: 9F03796, 0F03860, 3F04238, 4F04343
3. TECHNICAL REVIEWER: Dallas Wright, Jr.
4. DATE: June 19, 1995
5. ANALYTES/LEVEL: See Review
6. COMMODITIES: Pecans, Corn Grain, Corn Fodder, Soybean Seed, Oranges
7. METHOD: Determination of Residues of the Trimethylsulfonium Cation in Agricultural Crops by Gas Chromatography

The Analytical Chemistry Section has been asked to screen the residue chemistry methods submitted by the registrant in order to determine if they contain the essential requirements identified in the Residue Chemistry Guidelines. Full scientific review and laboratory evaluation of those methods will take place after the initial screen. The following items need to be resolved before the analytical method can be evaluated.

1. Does the method use exotic equipment and/or supplies that are not commercially available in the U.S.? YES NO
2. Does the method require any new equipment before the laboratory work begins? YES NO
3. Are chromatograms included?
   a. Is (are) peak(s) of interest sufficiently resolved from other peaks? YES NO
   b. Has registrant included chromatograms of analyses at or below tolerance on all crop types for which tolerance is requested by HED? See Review YES NO
c. Do the control samples have reasonably low levels of the analyte in relation to the proposed tolerance? YES NO
d. Is the method sufficiently sensitive and specific to measure and identify the residues at levels specified by HED in the TNV request? YES NO
4. Has recovery data been provided to ACL for the residues that are specified in the TMV request? Yes [ ] No [X]

5. Are recovery values between 70% and 120% at all levels and for all commodity types? Yes [ ] No [X]

6. Are all procedures clearly written with no ambiguities so that the method can be run without communication with the registrant? Yes [X] No [ ]

7. Does the method require correction for a sample of the untreated commodities or a blank? Yes [ ] No [X]

8. Does the method require the use of an internal or procedural standard to compensate for lost analyte during analysis? Yes [X] No [ ]

9. Are 2nd laboratory validation data provided with the method? Yes [ ] No [X]

10. Are there any deficiencies other than those covered above that would prevent ACS from conducting a method trial? Yes [X] No [ ]

11. Is this method suitable for validation testing? Yes [X] No [ ]

Any deficiencies/problems noted for any above items should be addressed in the full scientific review of this method to be attached as an addendum.

[Signature] 6/19/95 [Date]

The following is to be completed by the analyst performing the TMV.

12. Can a set of 6 samples be run within 24 hours? Yes [X] No [ ]

13. a. Are standards available at RTP repository? Yes [X] No [ ]

   b. Are derivatized analytical reference standards available? Yes [X] No [ ]
Linearity of TMS
GLC/FPD
9/11/95

<table>
<thead>
<tr>
<th>ug/mL</th>
<th>PK. HT. 1</th>
<th>PK. HT. 2</th>
<th>AVE PK. HT.</th>
<th>Sqrt Avg PK HT</th>
<th>Sqrt CALC. PK HT</th>
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<td>1285.0712</td>
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Regression Output:

Constant: -92.6310293
Std Err of Y Est: 104.8272386
R Squared: 0.964211424
No. of Observations: 4
Degrees of Freedom: 2
X Coefficient(s): 1148.08523
Std Err of Coef.: 156.4030464

Linearity of TMS
GLC/FPD 9/11/95
Linearity of TMS
GLC - FPD
7/5/95

<table>
<thead>
<tr>
<th>ug/mL</th>
<th>PK. HT 1</th>
<th>PK. HT 2</th>
<th>AVG PK. HT</th>
<th>CALC. PK. HT</th>
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<td>1.200</td>
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</table>

Regression Output:

- Constant: -317378.847826
- Std Err of Y Est: 228393.556228
- R Squared: 0.89778271334
- No. of Observations: 4
- Degrees of Freedom: 2
- Std Err of Coef.: 0.089136462
- X Coefficient(s): 1428058.609
- Std Err of Coef.: 340784.943

LINEARITY OF TMS
GLC/FPD 7/5/95

- Actual Data Points - Calculated Line

![Graph showing linearity with concentration (ug/ml) on the x-axis and peak height on the y-axis.](image-url)
RUN # 545    AUG 5, 1995  13:32:16
START

IF

IF

IF 1.163

TIMETABLE STOP

RUN# 545    AUG 5, 1995  13:32:16

SAMPLE NAME: DMSS STD    SAMPLE# 1
0.25 ug/ml

B95/34 THRU 39 <SOYBEAN SEED> 2UL INJ

HEIGHT X

RI  | HEIGHT TYPE WIDTH   HEIGHT X
   |   |                  
   1.163 | 49240 BU  .015 100.00000

TOTAL HEIGHT= 49240
MUL FACTOR=1.00000E+00

RUN PARAMETERS
ZERO = 20
ATT 2° = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WO = 0.02

RUN # 546    AUG 5, 1995  13:35:56
START

IF

IF

IF .215

TIMETABLE STOP

RUN# 546    AUG 5, 1995  13:35:56

SAMPLE NAME: CONTROL #1    SAMPLE# 2
FINAL VOL 1ML/50 ALIQUOT

B95/34 THRU 39 <SOYBEAN SEED> 2UL INJ

NO RUN PEAKS STORED

RUN PARAMETERS
RUN PARAMETERS
ZERO = 20
ATT 2" = 4
CHT SP = 1.0
AR REJ = 6000
THRESH = 3
PK WD = 0.02

RUN # 546 AUG 5, 1995 13:35:56
START

TIMETABLE STOP

RUN# 546 AUG 5, 1995 13:35:56
SAMPLE NAME: CONTROL #1
SAMPLE# 2
FINAL VOL IML/5G ALIQUOT

B95/34 THRU 39 (SOYBEAN SEED) 2UL INJ

NO RUN PEAKS STORED

RUN PARAMETERS
ZERO = 20
ATT 2" = 4
CHT SP = 1.0
AR REJ = 6000
THRESH = 3
PK WD = 0.02

RUN # 547 AUG 5, 1995 13:39:36
START

TIMETABLE STOP

RUN# 547 AUG 5, 1995 13:39:36
SAMPLE NAME: CONTROL #1
SAMPLE# 2
FINAL VOL IML/5G ALIQUOT

B95/34 THRU 39 (SOYBEAN SEED) 2UL TNT
RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRESH = 3
PK UD = 0.02

RUN # 554  AUG 5, 1995  14:05:18
START
IF
IF
IF 1.155
TIMETABLE STOP

RUN#  554  AUG 5, 1995  14:05:18
SAMPLE NAME: 0.05PAM SPK#  SAMPLE#  6
FINAL VOL 1ML/5G ALIQUOT

895/34 THRU 39 <SOYBEAN SEED> 2UL INJ
HEIGHTX
RT   HEIGHT  TYPE  WIDTH  HEIGHTX
1.155  40259  PU  .018  100.00000
TOTAL HEIGHT= 40259
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRESH = 3
PK UD = 0.02

RUN # 555  AUG 5, 1995  14:09:01
START
IF
IF
IF 1.155
TIMETABLE STOP

RUN#  555  AUG 5, 1995  14:09:01
SAMPLE NAME: 0.05PPM SPK#  SAMPLE#  6
FINAL VOL 1ML/5G ALIQUOT
TOTAL HEIGHT = 27393
MUL FACTOR = 1.000000E+00

RUN PARAMETERS
ZERO = 20
ATT 2\degree = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 560 AUG 5, 1995 14:27:22
START

IF

IF

'1.152 <
TIMETABLE STOP

RUN# 560 AUG 5, 1995 14:27:22

SAMPLE NAME: 0.5PPM SPK#2  SAMPLE# 9
FINAL VOL 10ML/5G ALIQUOT

B95/34 THRU 39 (SOYBEAN SEED) 2UL INJ

HEIGHT
RT  HEIGHT  TYPE  WIDTH  HEIGHT\%
1.152  34198  PV .017  100.00000

TOTAL HEIGHT = 34198
MUL FACTOR = 1.000000E+00

RUN PARAMETERS
ZERO = 20
ATT 2\degree = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 561 AUG 5, 1995 14:31:03
START

IF

IF

1.061
IF

1.152
RUN PARAMETERS
ZERO = 20
ATT 2\(^{\circ}\) = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 567 AUG 5, 1995 14:53:11
START

IF

IF

IF 1.148

TIMETABLE STOP

RUN# 567 AUG 5, 1995 14:53:11
SAMPLE NAME: 1PPM SPK#2 SAMPLE# 12
FINAL VOL 20ML/5G ALIQUOT

895/34 THRU 39 <SOYBEAN SEED> 2UL INJ

HEIGHT
RT HEIGHT TYPE WIDTH HEIGHT%
1.148 32041 PU .018 100.00000

TOTAL HEIGHT= 32041
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2\(^{\circ}\) = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 568 AUG 5, 1995 14:56:48
START

IF

IF

IF 1.146

TIMETABLE STOP
895/34 THRU 39 (CORN FODDER) 2UL INJ

HEIGHT

<table>
<thead>
<tr>
<th>RT</th>
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<th>WIDTH</th>
<th>HEIGHT%</th>
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<tbody>
<tr>
<td>1.174</td>
<td>84906</td>
<td>PB</td>
<td>.014</td>
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TOTAL HEIGHT = 84906
MUL FACTOR = 1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2° = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK UD = 0.02

RUN # 371 JUL 27, 1995 16:34:23
START

TIMETABLE STOP

RUN# 371 JUL 27, 1995 16:34:23
SAMPLE NAME: OMS STD SAMPLE# 7
0.25 UG/ML

895/34 THRU 39 (CORN FODDER) 2UL INJ

HEIGHT

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<td>PB</td>
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TOTAL HEIGHT = 84906
MUL FACTOR = 1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2° = 4
CHT SP = 1.0
RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 361 JUL 27, 1995 15:39:45
START

IF

IF

IF

TIMETABLE STOP

RUN# 361 JUL 27, 1995 15:39:45
SAMPLE NAME: CONTROL #1 SAMPLE# 2
FINAL VOL 2ML/56 ALIQUOT

B95/34 THRU 39 (CORN FODDER) 2UL INJ

NO RUN PEAKS STORED

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 362 JUL 27, 1995 15:45:10
START

IF

IF

IF

TIMETABLE STOP

RUN# 362 JUL 27, 1995 15:45:10
SAMPLE NAME: CONTROL #2 SAMPLE# 3
RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 367 JUL 27, 1995 16:12:25
START

TIMETABLE STOP

RUN# 367 JUL 27, 1995 16:12:25
SAMPLE NAME: 0.1 SPK#1 SAMPLE# 5
FINAL VOL 2ML/5G ALiquot

B95/34 THRU 39 (CORN FEEDER) JUL INJ

HEIGHT
RT  HEIGHT  TYPE  WIDTH  HEIGHT%  HEIgHT  TYPE  WIDTH  HEIGHT%  HEIgHT
1.121  685?  PU  .015  7.12342  1.172  69903  UB  .014  92.07658

TOTAL HEIGHT  96260
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 368 JUL 27, 1995 16:17:57
START

1.172

90
RUN # 373    JUL 27, 1995  16:45:17
START

TIMETABLE STOP

RUN # 373    JUL 27, 1995  16:45:17

SAMPLE NAME: 1PPM SPK#1    SAMPLE# 8
FINAL VOL 200ML/5G ALIQUOT

895/34 THRU 39 (CORN FODDER) 2UL INJ

HEIGHT

<table>
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<tr>
<td>1.176</td>
<td>07658</td>
<td>PB .014</td>
<td>100.0000</td>
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TOTAL HEIGHT= 87658
MUL FACTOR=1.0000C+00

RUN PARAMETERS
ZERO = 20
ATT 2° = 4
CHT SP = 1.0
AR REJ = 6000
THRESH = 3
PK WD = 0.02

RUN # 374    JUL 27, 1995  16:50:45
START

TIMETABLE STOP
RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRS = 3
PK WD = 0.02

RUN # 380 JUL 27, 1995 17:23:36
START
IF
IF
IF 1.100 <=

TIMETABLE STOP

RUN# 380 JUL 27, 1995 17:23:36

SAMPLE NAME: ZPPM SPK#2 SAMPLE# 12
FINAL VOL 40ML/5G ALIQUOT

B95/34 THRU 39 (CORN FODDER) 2UL INJ

HEIGHT RT HEIGHT TYPE WIDTH HEIGHT
1.100 76587 PB .013 100.00000

TOTAL HEIGHT = 76587
MUL FACTOR = 1.00000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 4
CHT SP = 1.0
AR REJ = 6000
THRS = 3
PK WD = 0.02

RUN # 381 JUL 27, 1995 17:29:03
START
IF
IF
IF 1.170
RUN PARAMETERS
ZERO = 20
ATT 2^ = 3
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 148 JUL 12, 1995 09:29:43
START

IF
IF 1.178

TIMETABLE STOP

RUN# 148 JUL 12, 1995 09:29:43
SAMPLE NAME: CONTROL#1 SAMPLE# 2
2UL INJ/FINAL VOL 1ML/56 ALIQUOT

B35-34 THRU 39 (CORN GRAIN)
NO RUN PEAKS STORED

RUN PARAMETERS
ZERO = 20
ATT 2^ = 3
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 149 JUL 12, 1995 09:35:12
START
HEIGHTy
 RT  HEIGHt  TYPE  WIDTH  HEIGHtX
 1.164  25518  'UU'  .016  100.00000

TOTAL HEIGHT = 25518
MUL FACTOR = 1.0000E+00

RUN PARAMETERS
ZERO  = 20
ATT 2^  = 3
CHT SP  = 1.0
AR REJ  = 6000
THRSH  = 3
PK WD  = 0.02

RUN # 154  JUL 12, 1995  10:02:31
START

IF
IF
IF 14957
       1.164 <

TIMETABLE STOP

RUN # 154  JUL 12, 1995  10:02:31

SAMPLE NAME: 0.05PPM SPK1  .SAMPLE#  5
2UL INJ/ FINAL VOL 1ML/ 5G ALIQUOT

895-34 THRU 39 (CORN GRAIN)

HEIGHty
 RT  HEIGHt  TYPE  WIDTH  HEIGHtX
 1.164  23776  'UU'  .015  100.00000

TOTAL HEIGHT = 23776
MUL FACTOR = 1.0000E+00

RUN PARAMETERS
ZERO  = 20
ATT 2^  = 3
CHT SP  = 1.0
AR REJ  = 6000
THRSH  = 3
PK WD  = 0.02
RUN PARAMETERS
ZERO = 20
ATT = 3
CHT SP = 1.0
AR REJ = 6000
THRSH = 3
PK WD = 0.02

RUN # 161 JUL 12, 1995 10:41:03
START

IF
IF
IF 1.163 ←

TIMETABLE STOP

RUN # 161 JUL 12, 1995 10:41:03

SAMPLE NAME: 0.1SPK1
SAMPLE: 0
ZUL INJ/ FINAL VOL2ML/ 5G ALIQUOT,

895-34'THRU 39(CORN GRAIN)

HEIGHT
RT  HEIGHT TYPE WIDTH  HEIGHT
1.163 23813 PU .016 100.0000

TOTAL HEIGHT= 23813
MUL FACTOR=1.0000E+00

RUN PARAMETERS
RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THREH = 3
PK WD = 0.06

RUN # 1217 Aug 29, 1995 10:54:36
START

TIMETABLE STOP

Storing processed peaks to A:Q14398EF.PRO

RUN# 1217 Aug 29, 1995 10:54:39
SAMPLE NAME: 0.25UG/ML

SAMPLE# 1

DMS STD

PEAK FILE : A:Q14398EF.PRO

B95/34-39 (PECANS) 2UL INJ

HEIGHT
RI  HEIGHT  TYPE  WIDTH  HEIGHT%
1.013  8814  BB  .015  6.79301
1.140  118403 BB  .022  91.25402
1.252  2534  BB  .016  1.95297

TOTAL HEIGHT= 129751
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THREH = 3
PK WD = 0.06

RUN # 1218 Aug 29, 1995 10:59:49
START

IF

j
RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRS = 3
PK WD = 0.06

RUN # 1232 AUG 29, 1995 12:34:18
START

IF

1.291
1.139 ←

TIMETABLE STOP

Storing processed peaks to R:Q143B04B.PRO

RUN# 1232 AUG 29, 1995 12:34:18

SAMPLE NAME: 0.05PPM SPK# SAMPLE# 5
FINAL VOL INML/5G ALIQUOT

PEAK FILE : R:Q143B04B.PRO

B95/34-39 (PECANS) 2UL INJ

HEIGHT%

RT HEIGHT TYPE WIDTH HEIGHT%
1.010 12107 PP .020 7.97369
1.088 9140 PP .014 6.01961
1.139 127017 PB .021 83.65354
1.251 3573 PB .023 2.35318

TOTAL HEIGHT= 151.037
MUL FACTOR=1.00000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRS = 3
PK WD = 0.06

RUN # 1233 AUG 29, 1995 12:39:39
START

IF

1.139

29
RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK UD = 0.06

RUN # 1239 AUG 29, 1995 13:11:46
START

IF

1.248

1.137 ≤

TIMETABLE STOP

Storing processed peaks to A:Q143B913.PRO

RUN# 1239 AUG 29, 1995 13:11:46
SAMPLE NAME: 0.1PPM SPK#1 SAMPLE# 8
FINAL VOL 2ML/5G ALIQUOT

PEAK FILE : A:Q143B913.PRO

B95/34-39 (PECANS) 2UL INJ

HEIGHT%

RT  |  HEIGHT  | TYPE | WIDTH | HEIGHT%
1.137 | 136306   | PB   | .022  | 97.70829
1.248 | 3197     | BB   | .013  | 2.29171

TOTAL HEIGHT= 139503
MUL FACTOR=1.00000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK UD = 0.06

RUN # 1240 AUG 29, 1995 13:17:06
START

IF

1.000

TIMETABLE STOP
TOTAL HEIGHT = 251509
MUL FACTOR = 1.00000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

RUN # 1075 AUG 19, 1995 13:40:50
START

TIMETABLE STOP

Storing processed peaks to A:Q13690E3.PRO

RUN# 1075 AUG 19, 1995 13:40:50
SAMPLE NAME: 0.25UG/ML
DMS STD
PEAK FILE: A:Q13690E3.PRO
B35/34-39 <ORANGES> 2UL INJ

<table>
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<td>118788</td>
<td>UV</td>
<td>.021</td>
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<td>1.245</td>
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<td>2.203</td>
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<td>PP</td>
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TOTAL HEIGHT = 244615
MUL FACTOR = 1.00000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
**RUN # 1064**

**AUG 19, 1995 12:36:40**

**START**

- **IF**
  - $0.865_{1.243}$
  - **1.660**

**TIMETABLE STOP**

Storing processed peaks to A:Q1368109.PRO

**RUN# 1064**

**AUG 19, 1995 12:36:40**

**SAMPLE NAME: CONTROL#1**

**SAMPLE# 2**

**FINAL VOLIML/5G ALIQUOT**

**PEAK FILE : A:Q1368109.PRO**

**B95/34-39 (ORANGES) 2UL INJ**

**HEIGHT**

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<td>.142</td>
<td>1.65205</td>
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<td>2.214</td>
<td>1974</td>
<td>.039</td>
<td>1.03490</td>
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</table>

**TOTAL HEIGHT = 142429**

**NUL FACTOR = 1.0000E+00**

**RUN PARAMETERS**

- **ZERO** = 20
- **ATT 2** = 5
- **CHT SP** = 1.0
- **AR REJ** = 2000
- **THRSH** = 3
- **PK UD** = 0.04

**RUN # 1065**

**AUG 19, 1995 12:42:32**

**START**

- **IF**
  - $0.891_{1.295}$
  - **1.665**

**TIMETABLE STOP**
TOTAL HEIGHT= 109984  
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2° = 5
CHI SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

RUN # 1071  AUG 19, 1995  13:17:02  
START

IF
0.731
1.250
1.168
1.656
2.209

TIMETABLE
STOP

Storing processed peaks to A:Q1368B4F.PRO

RUN# 1071  AUG 19, 1995  13:17:02

SAMPLE NAME: 0.05PPM  SPK#  SAMPLE#  S  
FINAL VOL 1ML/50 ALIQUOT

PEAK FILE : A:Q1368B4F.PRO

895/34-39 (ORANGES) 2UL INJ

HEIGHT

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TOTAL HEIGHT= 244298  
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2° = 5
CHI SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

33
RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

RUN # 1091   AUG 19, 1995  14:57:23
START

IF

0.537

.13392  1.156  ←

TIMETABLE STOP

.1645

 Sto ring processed peaks to A:Q136A204.PRO

RUN# 1091   AUG 19, 1995  14:57:23
SAMPLE NAME: 0.5PPM SPK#1: SAMPLE# 0
FINAL VOL 10ML/50 ALIQUOT
PEAK FILE : A:Q136A204.PRO
B95/34-39 (ORANGES) 2UL INJ

HEIGHT
RT  HEIGHT TYPE  WIDTH  HEIGHT%
.537  1896 BP  .110  .79338
1.112  13816 PU   .017  5.61418
1.156  75239 UU  .023  31.72165
1.239  11545 UU  .023  4.06751
1.645  135189 BP  .122  56.99728

TOTAL HEIGHT= 237185
MUL FACTOR=1.0000E+00

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CHT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

RUN # 1092   AUG 19, 1995  15:01:23
START

IF
0.460

34
NO RUN PEAKS STORED

RUN PARAMETERS
ZERO = 20
ATT 2^ = 5
CMT SP = 1.0
AR REJ = 2000
THRSH = 3
PK WD = 0.04

* EDIT SEQ

1 = ALS INFORMATION
2 = EQUILIBRATION TIME DELAY
3 = METHOD FILE SPECIFICATION
4 = SAMPLE INFORMATION TABLE

SECTION TO BE EDITED: 1

ALS INFORMATION
INET SAMPLER CONTROL [Y*/N]:

7673A SAMPLER:
LOOP ADDRESS: 0

FRONT INJECTOR
INJ/BOottle 2 --> @
FIRST BOTTLE 7 --> 12 @
LAST BOTTLE 13 --> BREAK

* SEQ START
RUN # 1099 AUG 19, 1995 15:27:39
START

TIMETABLE STOP

Storing processed peaks to A:Q136A9EC.PRO

RUN# 1099 AUG 19, 1995 15:27:39

SAMPLE NAME: 1PPM SPK#2 SAMPLE# 12
FINAL VOL 20mL/5g ALIQUOT

PEAK FILE : A:Q136A9EC.PRO

095/34-39 (ORANGES) 2UL INJ

HEIGHTX
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TOTAL HEIGHT 212360